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ENGINEERING SERVICE CENTER
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METHOD FOR DETERMINATION OF PIGMENTS AND EXTENDERS IN PAINTS AND COATINGS

CAUTION: Prior to handling test materials, performing equipment setups, and/or conducting this method, testers are required to read "**SAFETY AND HEALTH**" in Section E of this method. It is the responsibility of the user of this method to consult and use departmental safety and health practices and determine the applicability of regulatory limitations before any testing is performed.

A. SCOPE

This method describes the X-ray diffraction procedure used for analysis of paint pigments.

B. APPARATUS

1. X-ray diffractometer with direct recording attachments.
2. Opacity Charts, Leneta Form 2A, are available from Leneta Company, P. O. Box 86, Ho-Ho-Kus, NJ 07423. They are used as substrates for the paint films. Other materials may be satisfactory provided they do not contribute interfering diffraction peaks to the sample pattern.

C. SAMPLE PREPARATION

1. Liquid Samples:
 - a. Apply a uniform film of the sample to the glazed side of the Leneta Chart and allow to dry thoroughly. (Coating applicator blades in various depths are useful in achieving a uniform film of the sample. Experience will indicate the proper film thickness for satisfactory determinations).
 - b. After the coating has dried, a suitably sized specimen may be cut from the chart to fit into the X-ray diffractometer.
2. Dry Pigments and/or Extender Pigments:

- a. Extract pigments as specified in Section D-2 of California Test 402 are required. Pass powdered pigments and extender pigments through a standard 300- μ m sieve.
- b. Prepare the powder sample for diffraction as instructed in the manual used for the X-ray diffraction apparatus.

D. TEST PROCEDURE

1. Using a stiff material such as a glass microscope slide to support the dried specimen from behind, insert the specimen into the diffractometer and run a diffraction scan covering a "d" range of 22-1.54 Å.
2. Adjust the diffractometer initially to produce a recorder deflection of about 70 % for the strongest diffraction peak in the pattern. Further adjustments may be necessary to increase the intensities of weak or poorly diffracting materials.
3. While standard tables are available for identifying peaks, best results are obtained by comparison with diffraction curves of standard paints of known compositions that have been prepared under controlled conditions in the laboratory. It is possible to determine which pigments and extenders are present and to obtain some indication of the relative amounts.
4. When a pigment or extender appears to be absent or in the wrong percentage by X-ray diffraction, it is necessary to confirm the absence by X-ray emission, wet chemical, or petrographic analysis.

Petrographic analysis may be used for confirming the presence of diatoms from the extender pigment, diatomaceous silica— when there is not sufficient crystallinity to give an X-ray diffraction pattern.

5. Reference numbers 2, 3, or 4 may be used to find the percent of the elements present in the pigment, which may be then analyzed chemically by one of the following methods to determine if the elements are present in the required amount.
 - a. ASTM Designation: D 717, Section 2.a-c, SiO_2 .
 - b. ASTM Designation: C 114, Sections 9 to 15, Fe_2O_3 , Al_2O_3 , CaO , MgO (after preliminary sample preparation by ASTM Designation: D 717). Fe_2O_3 should be done on duplicate sample by re-dissolving the R_2O_3 precipitate.
 - c. Scott's Standard Method of Chemical Analysis, Volume II, Fifth Edition, Page 883, "Hydrofluoric Acid Method for Alkalis in Silicates" and ASTM Designation: C 114, Section 55 are available. However, it will not be necessary to use CaCl_2 stock solution to prepare the standard. Method for determining Na_2O and K_2O may be used.

E. SAFETY AND HEALTH

This method may involve hazardous materials, operations, and equipment. This method does not purport to address all the safety problems associated with its use. It is the responsibility of whoever uses this method to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Observe good hygiene practices. Wash hands after handling samples and before eating, drinking or smoking.

X-ray producing equipment can be dangerous to both the operator and persons in the immediate vicinity unless safety precautions are strictly observed. Refer to the manufacturer's instruction manual. Exposure to excessive quantities of X-radiation may be injurious to health. Therefore, users should avoid exposing any parts of their bodies, not only to the direct beam, but also to secondary or scattered radiation that occurs

when an X-ray beam strikes or has passed through any material. It is strongly recommended that users check the degree of exposure by film carried on them or by the use of dosimeters and that blood counts be made periodically. Before utilizing the equipment, all persons designated or authorized to operate X-ray instrumentation or supervise its operation, should have a full understanding of its nature and should also become familiar with established safe exposure factors by a careful study of the National Institute of Standards and Technology Handbook "X-ray Recommendations of the International Roentgen Ray Committee on X-ray Protection", and other standard publications on the subject. Inquiries should be made of state agencies as to existing requirements.

Place colorful signs displaying the international radiation symbol near X-ray equipment.

When X-ray equipment is producing radiation, illuminate a conspicuous light.

There should be no X rays if the bulb burns out. Equipment without this feature can be modified.

Use a portable counter periodically to test for leakage of X rays from equipment. Lead or lead glass shielding is sometimes needed. X rays of shorter wavelength require more caution.

Prior to handling, testing or disposing of any waste materials, testers are required to read: Part A (Section 5.0), Part B (Sections: 5.0, 6.0, 10.0 and 12.0) and Part C (Section 1.0) of Caltrans Laboratory Safety Manual. These sections pertain to requirements for general safety principles, standard operating procedures, protective apparel, disposal of materials and how to handle spills, accidents, emergencies, etc. Users of this method do so at their own risk.

REFERENCES:

Optical Mineralogy, by Rogers & Ker-McGraw-Hill Book Company
Caltrans Standard Specifications
Dana's Manual of Mineralogy, 17th Edition, Revised by Hulburt, Jr.; John Wiley & Sons, Inc., Publisher
Organic Coating Technology, Vol. II, by Payne; John Wiley & Sons, Inc., Publishers
ASTM Designations: D 717 and C 114
California Test 402
Scott's Standard Method of Chemical Analysis, Volume II, Fifth Edition

End of Text (California Test 421 contains 2 pages)